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NAVAIR - SPONSORED AEROSOL RESEARCH AT  
NAVWPNSUPPCEN, CRANE - FY 79.

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Crane, Indiana 47522

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22 September 1980



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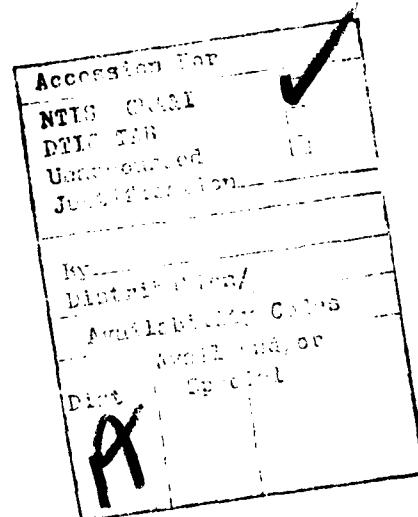
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-use; (2) 10.6 micrometer transmittance measurements on boron carbide ( $B_4C$ ) powder; (3) similar measurements on silicon carbide (SiC) powder; (4) attempts to prepare large quantities of <1 micrometer diameter olivine powder; (5) initial attempts to make olivine smoke by pyrotechnic means; and (6) progress toward modification and renovation of an existing cloud chamber to make a large aerosol chamber.

Using the disseminator to disseminate 2-3 micrometer  $B_4C$  and 2-3 micrometer SiC, the 10.6 micrometer extinction coefficient of both was shown to be  $.37 m^2 g$ . The reported value indicated that  $B_4C$  and SiC offer no improvement over the currently used aerosol screening material, phosphorus. Production of submicrometer size olivine powder ( $Mg_2SiO_4$ ) by sieving and ball milling was abandoned after repeated careful attempts ended with the material solidly caked on the walls of the container.

Magnesium silicide ( $Mg_2Si$ ) was incorporated in varying amounts with either sodium nitrate or potassium perchlorate into 10 g pyrotechnic candles, with the goal of producing a smoke containing submicrometer olivine particles. None of the compositions produced enough smoke to be effective.

The apparatus used in the  $B_4C$  and SiC experiments has been moved to a much more suitable location--one that is especially dedicated to aerosol experiments. This will make subsequent experiments much easier to perform.



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## PREFACE<sup>†</sup>

I would like to acknowledge the help of Ms. Theresa Drehobl and Mr. Forrest Burton in setting up and performing some of these experiments. Also, I appreciate the expert help of Mr. Bill Humerickhouse in the design and construction of the dry-powder disseminator.

<sup>†</sup>In order to specify procedures adequately, it has been necessary occasionally to identify commercial materials and equipment in this report. In no case does such identification imply recommendation or endorsement by the Navy, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

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## 1. INTRODUCTION

This report discusses aerosol infrared extinction measurements done by Naval Weapons Support Center (NAVWPNSUPPCEN), Crane for the Naval Air Systems Command (AIR-31OC) during FY 79. The purpose is to form the technological basis for development of new infrared screening aerosols with some emphasis on extinction at 10.6 micrometers. Subjects covered include (1) the completion and checkout of an aerosol dry powder disseminator for laboratory use, (2) 10.6 micrometer transmittance measurements on boron carbide ( $B_4C$ ) powder, (3) similar measurements on silicon carbide (SiC) powder, (4) attempts to prepare large quantities of < 1 micrometer diameter olivine powder, (5) initial attempts to make olivine smoke by pyrotechnic means, and (6) progress toward modification and renovation of an existing cloud chamber to make a large aerosol chamber.

## 2. AEROSOL DRY POWDER DISSEMINATOR

The design of this disseminator is based on existing units at Chemical Systems Laboratory, Aberdeen Proving Ground, Maryland. Figures 1-8 show the drawings. Figure 1 is an overall schematic of the unit. In general, it consists of the body which contains the dry powder, a screw-on cap, a set of air-flow tubes, an orifice (on the top of the cap), and a deflector cone (on the top of the cap above the orifice outlet). The unit is intended to function as follows:

Dry powder is placed in the body, filling to just below the air-flow tube outlets. The cap is screwed on. Compressed air is suddenly introduced into the nozzle. The air issues out the ends of the air-flow tubes and is directed down and to the opposite side of the body, causing extreme agitation of the powder. Simultaneously, air issues from four small horizontal holes in the nozzle (Section A-A in Figure 7). This air serves to help "lift" the dry powder so it can be better agitated by the air from the air-flow tubes. After the powder is agitated, it is forced up through the orifice (Figure 5) and impinges on the deflector (Figure 4). The deflector is intended to break up agglomerates in the powder.

FIGURE 1

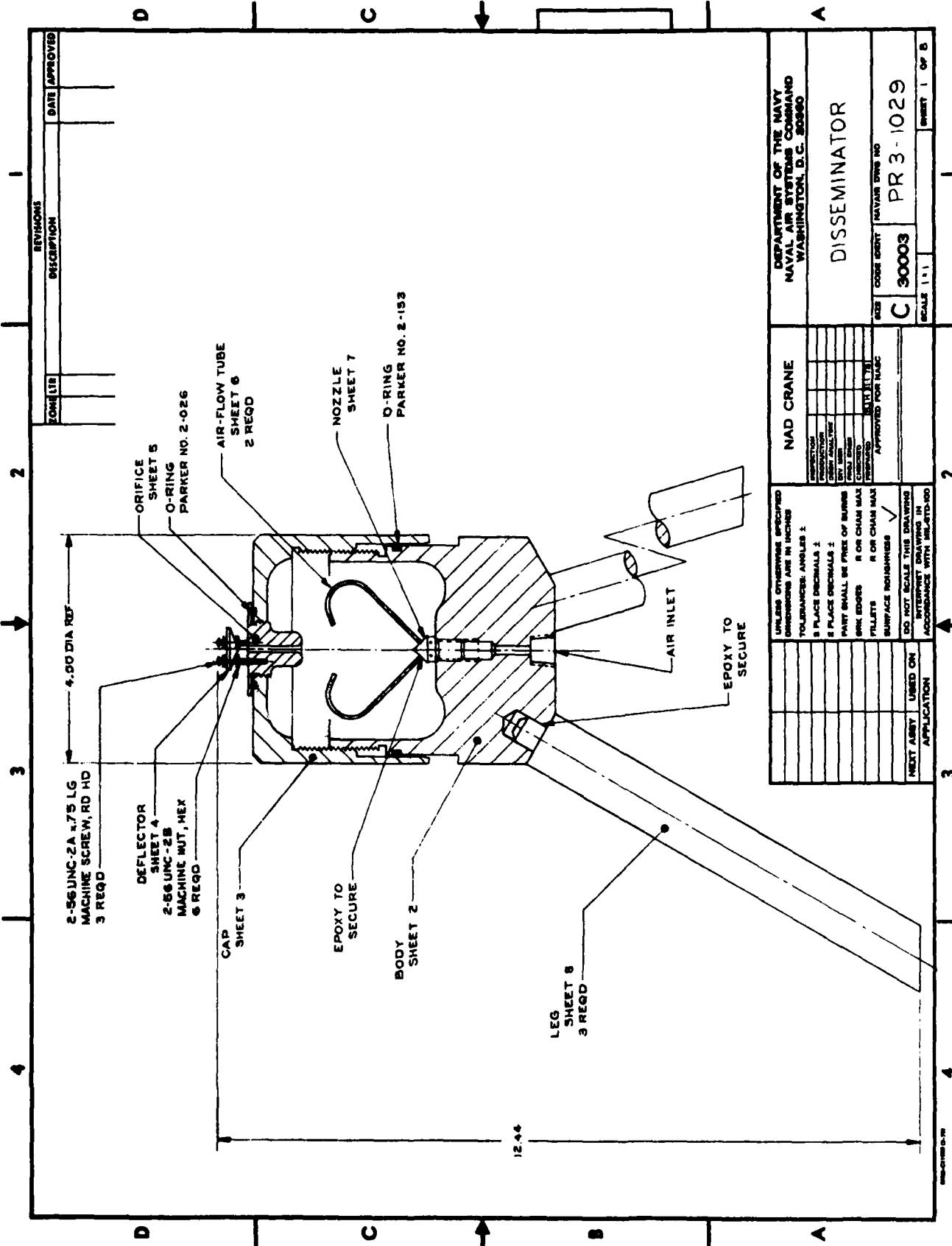


FIGURE 2

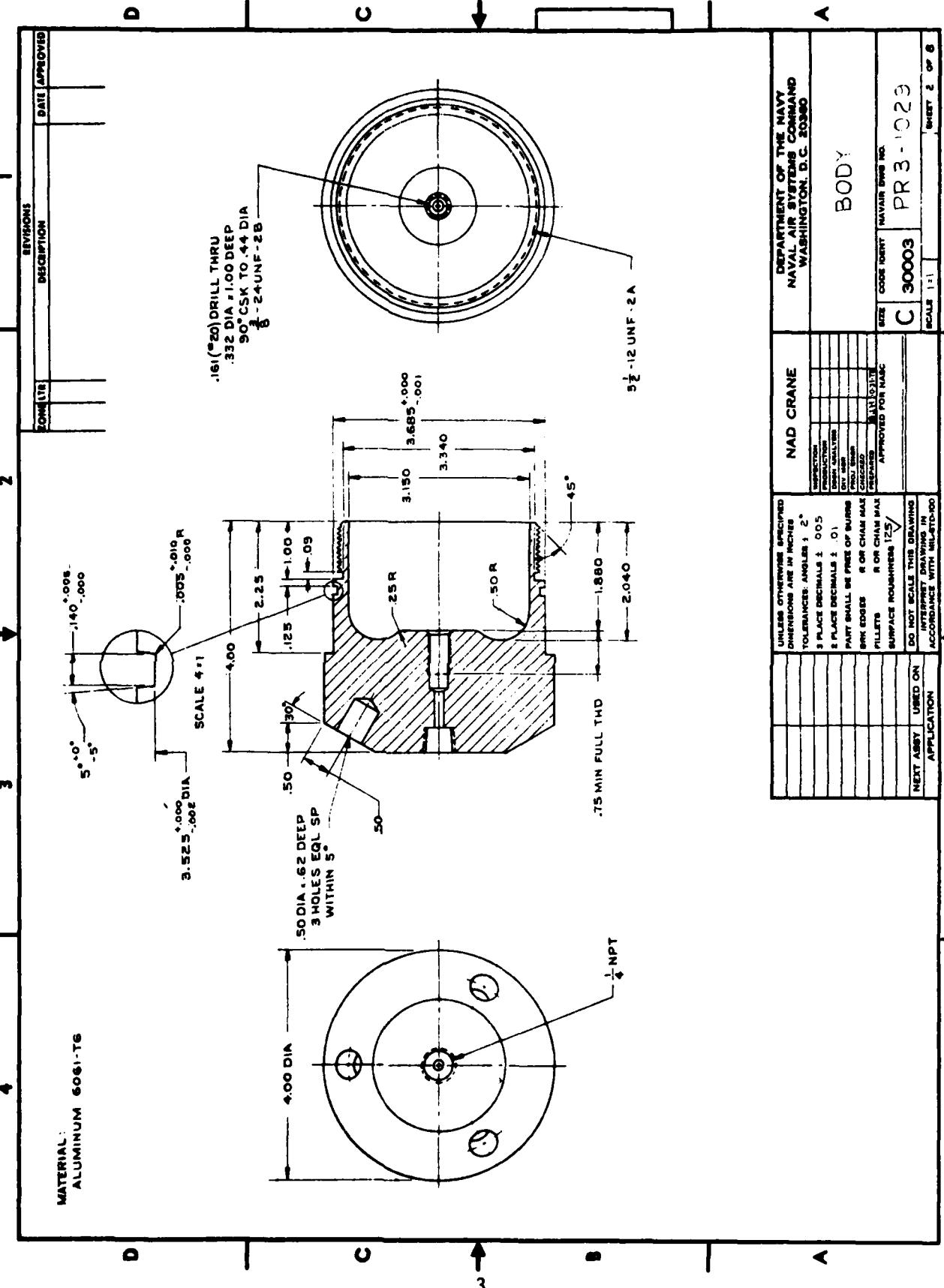


FIGURE 3

MATERIAL: ALUMINUM 6061-T6

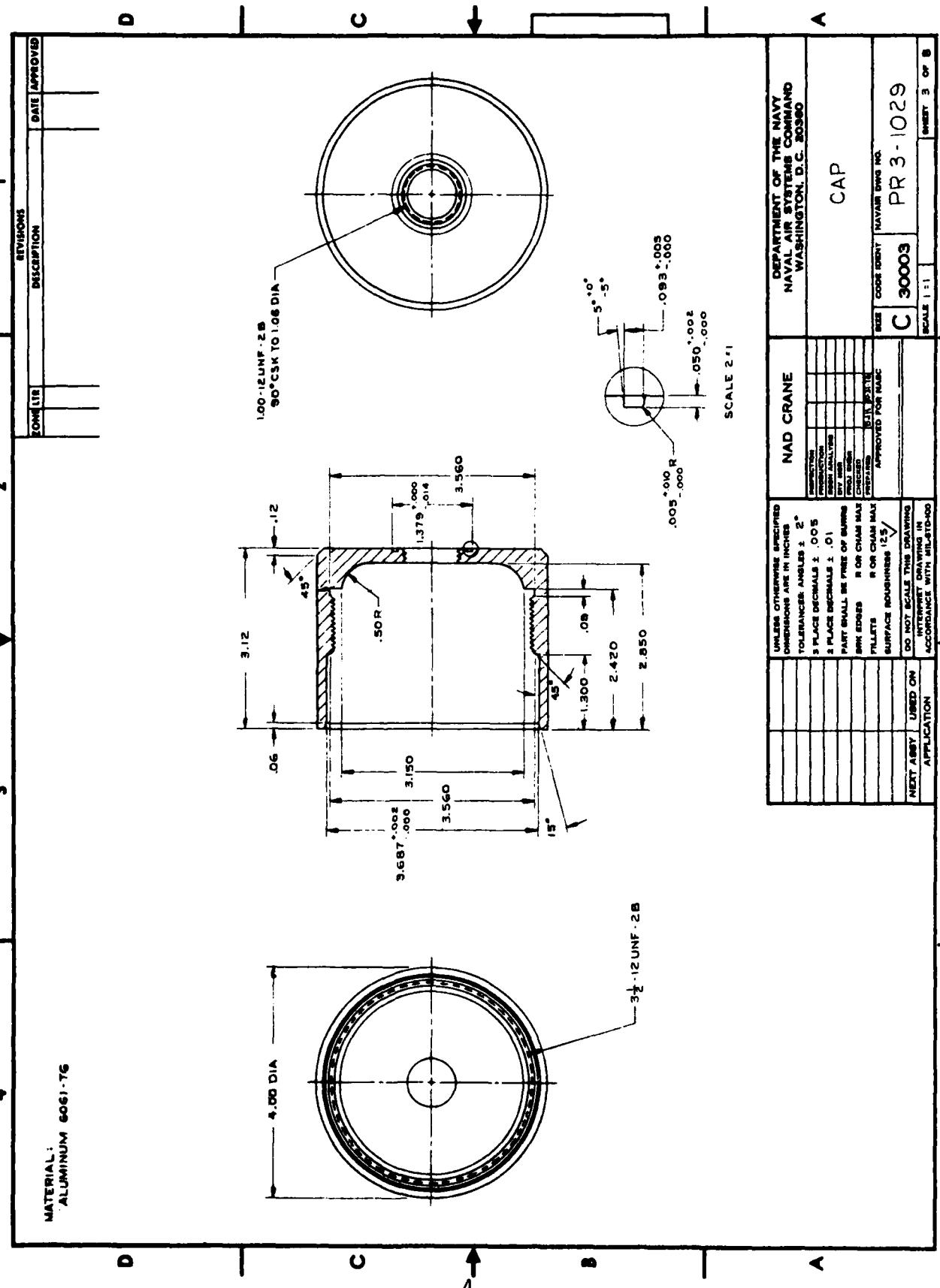
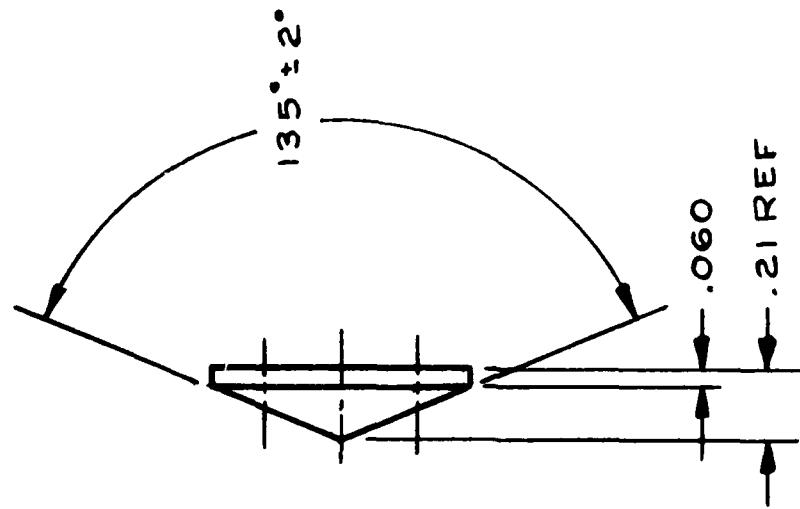
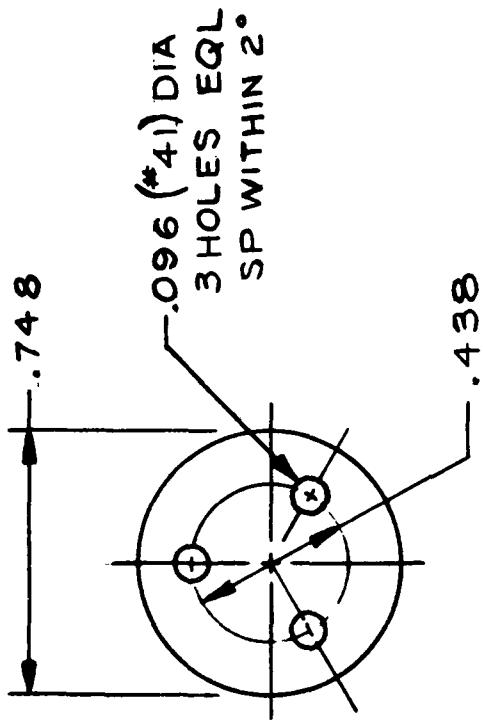


FIGURE 4



DEFLECTOR

PR 3-1029 SHEET 4-8

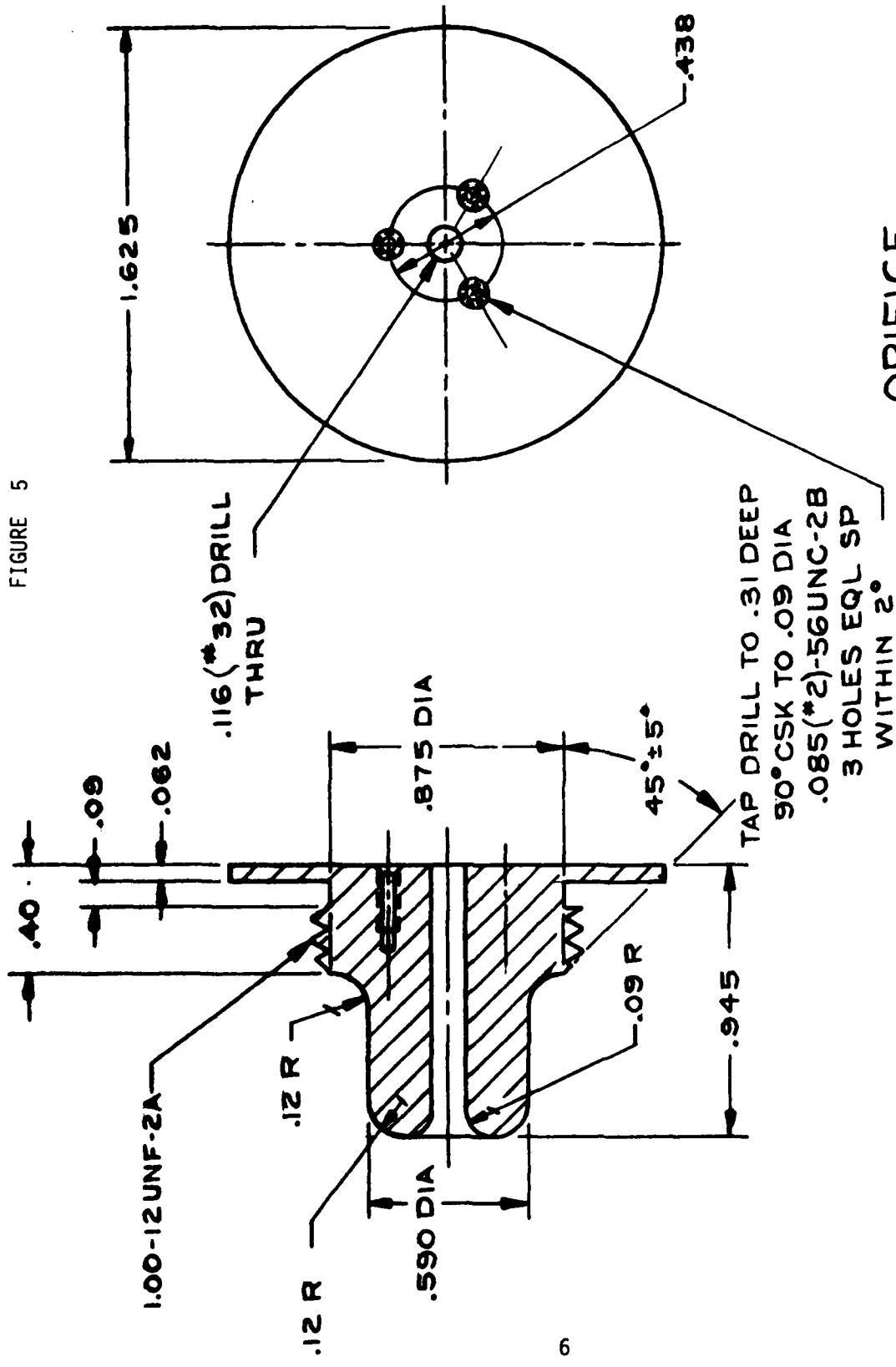
MATERIAL: ALUMINUM 6061-T6

OR 7075-T6

TOLERANCES: ±.005 ±.01

SCALE: 2 = 1 BJD 10-31-78

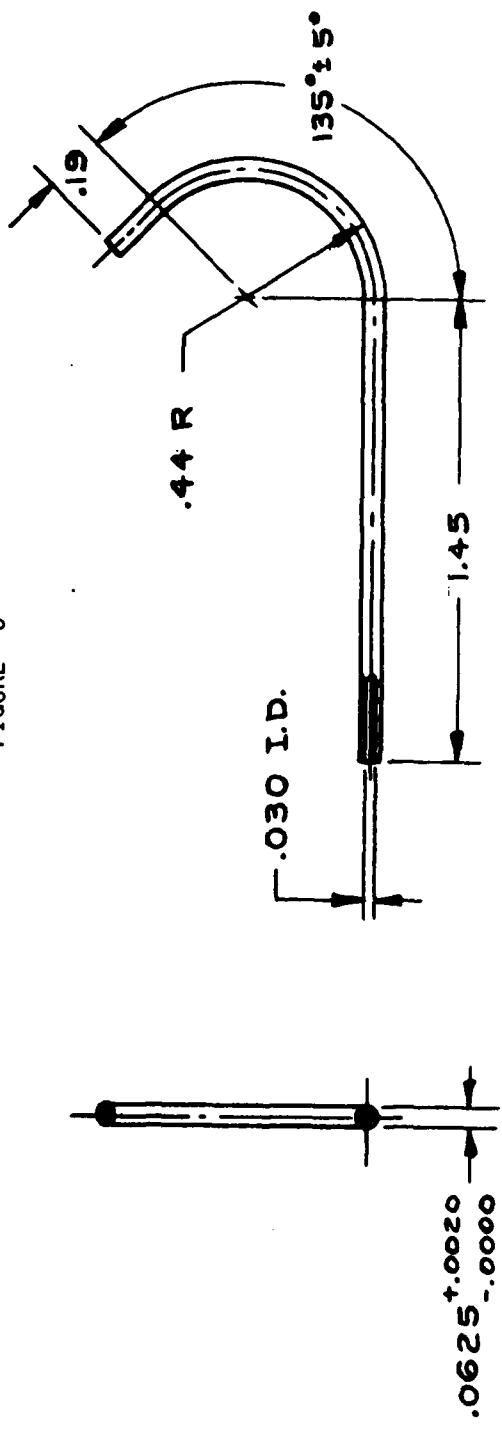
FIGURE 5



PR 3-1029 SHEET 5 or 8

MATERIAL: ALUMIN 6061-T6 or  
TOLERANCES:  $\pm .005 \pm .01$  7075-T6  
SCALE 2=1 BUH 10-31-78

FIGURE 6

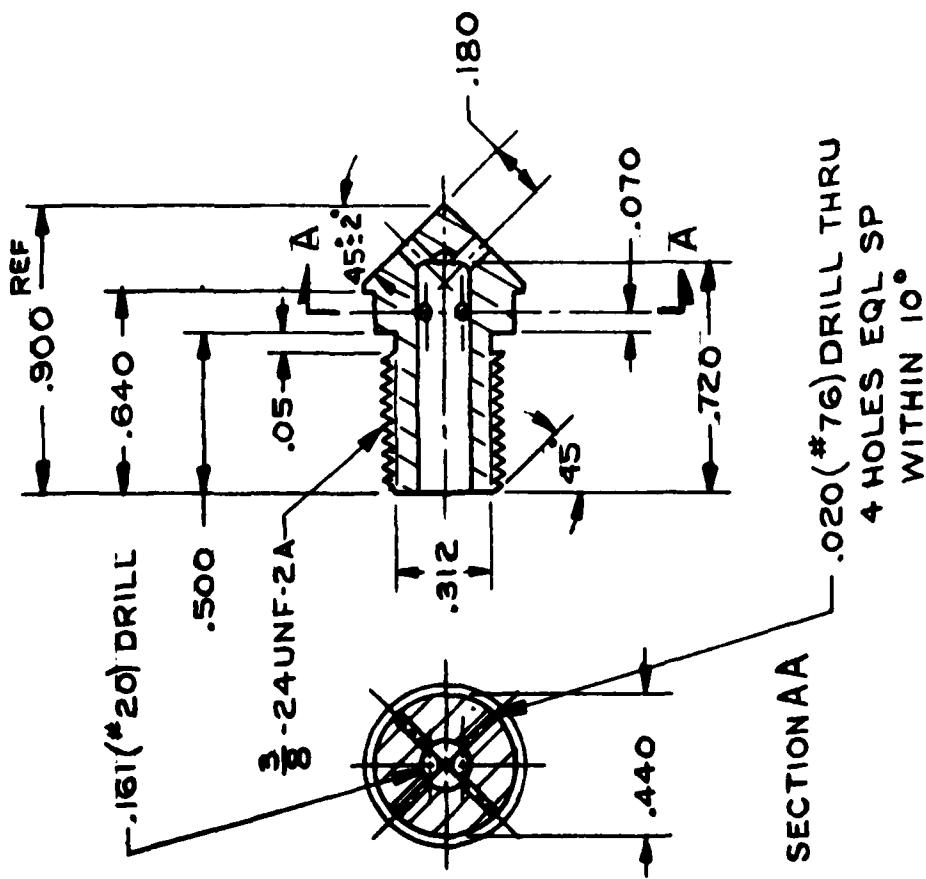


AIR-FLOW TUBE  
PR 3-1029- SHEET 6 of 8  
MATERIAL: STAINLESS STEEL,  
CAPILLARY TUBING  
TOLERANCES:  $\pm .005 \pm .01$   
SCALE: 2=1 B.U.H 10-31-78

MATERIAL SOURCE:  
SMALL PARTS INC.  
6901 N.E. THIRD AVE  
MIAMI, FLA 33138  
(PART # C7X-6230)

FIGURE 7

(This nozzle has since been modified by drilling 2 more .0635 holes at right angles to the original 2.)



## NOZZLE

PR 3-1029 SHEET 7 of 8

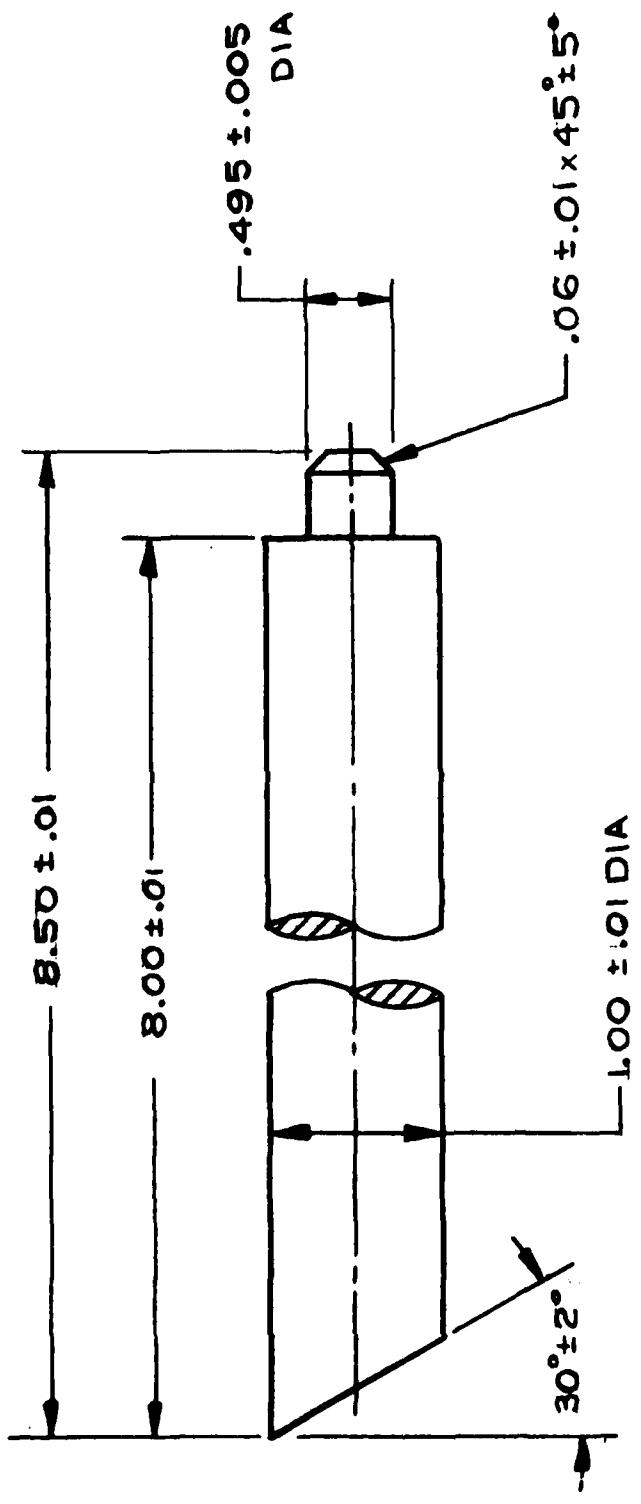
MATERIAL: ALUMIN 6061-T6 OR

7075-T6

TOLERANCES:  $\pm .005 \pm .01$

SCALE: 2:1 B.J.H 10-31-78

FIGURE 8



LEG

PR 3-1029 SHEET 8 OF 8  
MATERIAL: ALUMINUM 6061-T6 or  
7075-T6

SCALE: 1 = 1 B.J.H. 10-31-78

The unit is of modular design, so that different sized or shaped deflectors, orifices, nozzles, and air-flow tubes could be substituted. In the checkout (using silica powder), it was found that a number of modifications had to be made to get better dissemination and to make the unit easier to use. The nozzle was redesigned to accept four, instead of two, air-flow tubes to provide turbulence over more of the powder. The air-flow tubes were bent downward a bit more than called for in the original fabrication drawing (Figure 6) in order to better direct the air. The large O-ring (intended as a pressure seal) was removed because it made it very difficult to thread the cap on and off. Its absence did not cause any problems. A solenoid-operated air valve was placed in the air line as close as possible to the disseminator, in order to be able to introduce the air (at 100 pounds per square inch (psi)) with an instant "wallop" for maximum turbulence. Figure 9 shows the modified disseminator (with cap off). The air valve is at the left (covered in a plastic bag). The air hose has a quick disconnect fitting. Figure 10 is an overall view of the assembled disseminator on top of a rectangular storage box. The box is attached to a ballast tank which is filled to 100 psi from compressed air sources.

In use, the disseminator produces a cloud which can fill an approximate 4 x 6 x 4 meter (m) volume with different concentrations of aerosol, depending on the number of times the solenoid is opened. (A number of short bursts was found to be better than one long burst.) Problems remaining with the unit are static electricity buildup (a grounding strap doesn't help) and incomplete deagglomeration. At present no attempt has been made to optimize the orifice shape, deflector shape, or to mount anti-static units inside the cap, etc.

### 3. BORON CARBIDE ( $B_4C$ ) EXTINCTION COEFFICIENT AT 10.6 MICROMETERS

The extinction coefficient of boron carbide (type Norbide MCA 1482 1000F, Norton Company, Worcester, Massachusetts) of about 2-3 micrometer particle diameter, according to the manufacturer, was measured using the disseminator described above. This is the smallest commercially available  $B_4C$ . Figure 11 shows a scanning electron micrograph of boron carbide particles. The bar at the bottom represents one micrometer.

FIGURE 9  
DISSEMINATOR CLOSEUP



FIGURE 10  
DISSEMINATOR OVERALL VIEW

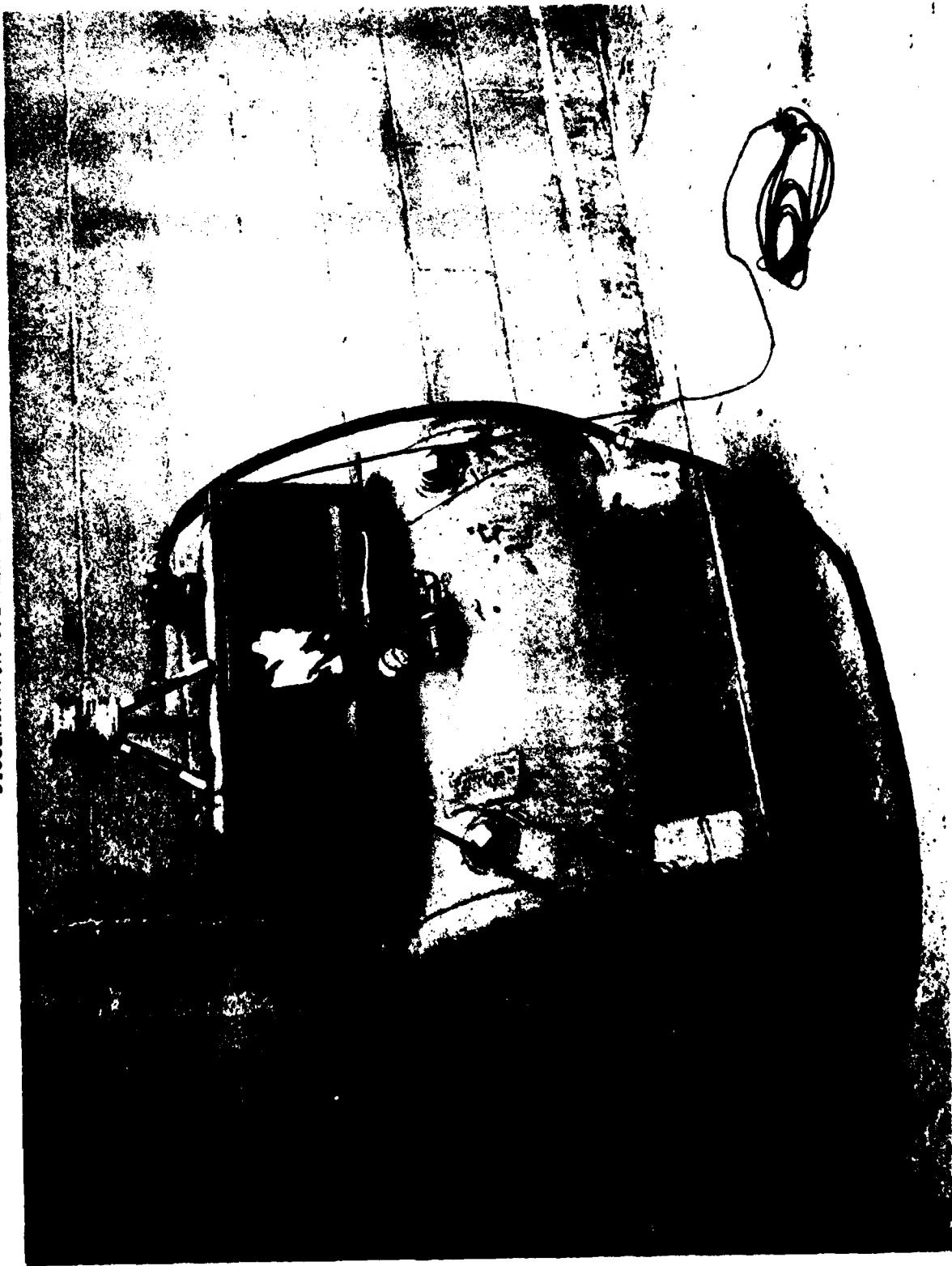


FIGURE 11  
SCANNING ELECTRON MICROGRAPH OF BORON CARBIDE PARTICLES



The basic equation used is the following:

$$\alpha = -\frac{\ln \tau}{cL} \quad (1),$$

where  $\tau$  is the transmittance;  $c$  is the concentration, in  $\text{g}/\text{m}^3$ ;  $L$  is the pathlength, in m; and  $\alpha$  is the attenuation coefficient, in  $\text{m}^2/\text{g}$ .

Figure 12 shows the experimental arrangement. This "chamber" is part of the photometric tunnel normally used for flare radiation measurements, but was converted to an aerosol chamber by using two plastic curtains at A.

A 3-watt cw  $\text{CO}_2$  Laser (Model 941, GTE Sylvania, Mountain View, California) producing a 4 milliradian (mrad) divergent,  $\text{TEM}_{00}$ , vertically polarized beam is located at B, in a room adjoining the chamber. The beam diameter at the laser was .45 centimeters (cm) and the beam diameter at the detector was 2 cm. The beam was not focused, nor were any apertures used. The thermopile detector (Model 201, Coherent Radiation, Palo Alto, California) is located at C. The detector surface is circular with a 1.9 cm diameter, and is recessed 3.7 cm inside a conical tube opening up to 2.5 cm diameter. Although there is no detector window, no evidence was ever found for deposition of aerosol on the detector active surface.

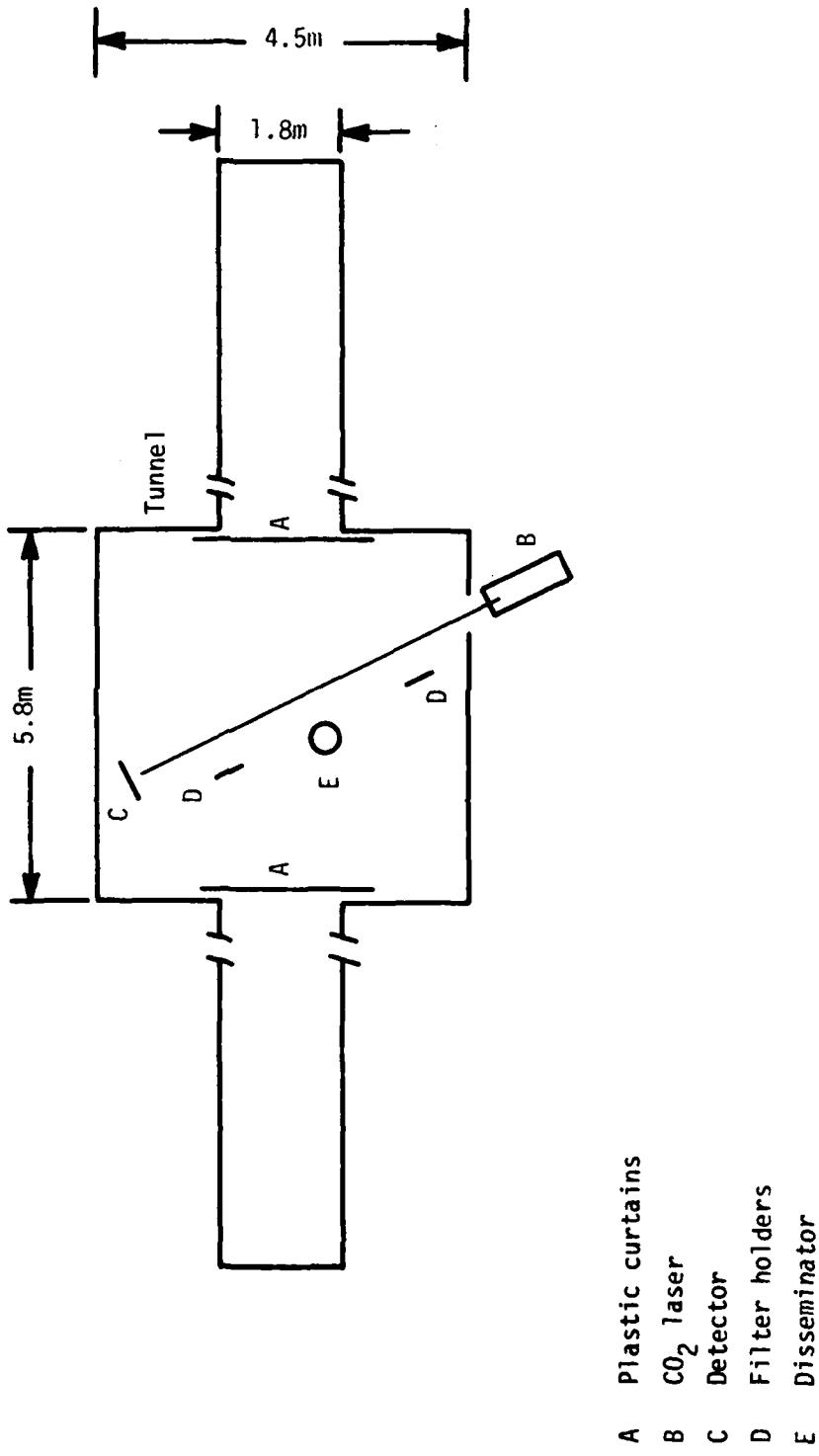
Using these laser beam and detector parameters, the laser wavelength, and the particle size of the boron carbide, calculations were done based on information in the literature<sup>1</sup> to determine the extent of corrections for forwardscattering in determining the extinction coefficient. The equations assume a homogeneous aerosol composed of spherical particles. The corrections, however, were found to be negligible.

Referring again to Figure 11, 4.7 cm inside diameter (ID) circular filter holders (Millipore Corporation, Bedford, Massachusetts) are located at D.

---

1. Deepak, A. and Box, M. A., Forwardscattering Corrections for Optical Extinction Measurements in Aerosol Media. 1: Monodispersions, Appl. Opt. 17 (18), 2900 (September 1978).

FIGURE 12  
EXPERIMENTAL ARRANGEMENT IN PHOTOMETRIC TUNNEL  
(TOP VIEW, NOT TO SCALE)



They each have a one liter/minute limiting orifice. Pairs of Millipore® type AA filters (.8 micrometer pore size) were used in some runs, and pairs of type VC filters (.1 micrometer pore size) were used in other runs. The filter holders were connected by equal lengths of plastic tubing through a "Y" joint to the vacuum pump and flow meter (not shown). The entire system was carefully checked to ensure there were no leaks. The filter holders were spaced symmetrically about 110 cm from the ends of the optical axis (3.9 m pathlength), at the same height as the optical axis, and about 15 cm offset from it. The lines perpendicular to the optical axis and passing through the centers of the filters coincided with the normal to the filters. Two filters, rather than one, were used in order to determine the extent of inhomogeneities in the aerosol concentration along the optical path. The disseminator was located at E.

The sequence of operations during a single run included the following:

- Load disseminator with  $B_4C$  powder
- Weigh two clean filters (to  $\pm .0002$  g)
- Record Temperature, Relative Humidity
- Load two filters into respective filter holders
- Unblock laser
- Record baseline detector signal (no aerosol in path)
- Open solenoid valve on disseminator for a single or for multiple bursts
- Record detector signal versus time until aerosol begins to dissipate
- Ventilate chamber
- Block laser
- Unload filters
- Weigh filters
- Record Temperature, Relative Humidity

For the  $B_4C$  runs, the weights of material on the filter papers were very low, such that the weighing errors were a significant fraction of the weights of  $B_4C$  on the filters. This effect masked any evidence of inhomogeneities in the aerosol concentration along the optical path. The reasons for this were subsequently determined, and will be explained below.

Figure 13 indicates the results. Essentially,  $\alpha$  is the slope of the least squares fit of the data. This relationship comes directly from equation (1). The transmittance is derived from the ratio of the recorded detector signal, with and without smoke in the optical path. The concentration  $c$  is derived from the total weight of  $B_4C$  on the two filters, divided by the number of  $m^3$  of air drawn through the filters. The concentration, in  $g/m^3$ , is multiplied by the path length  $L$ , in  $m$ , to give  $cL$  in  $g/m^2$ . The extinction coefficient  $\alpha$  is about  $.37\ m^2/g$ , but as is obvious, there is considerable scatter in the data.

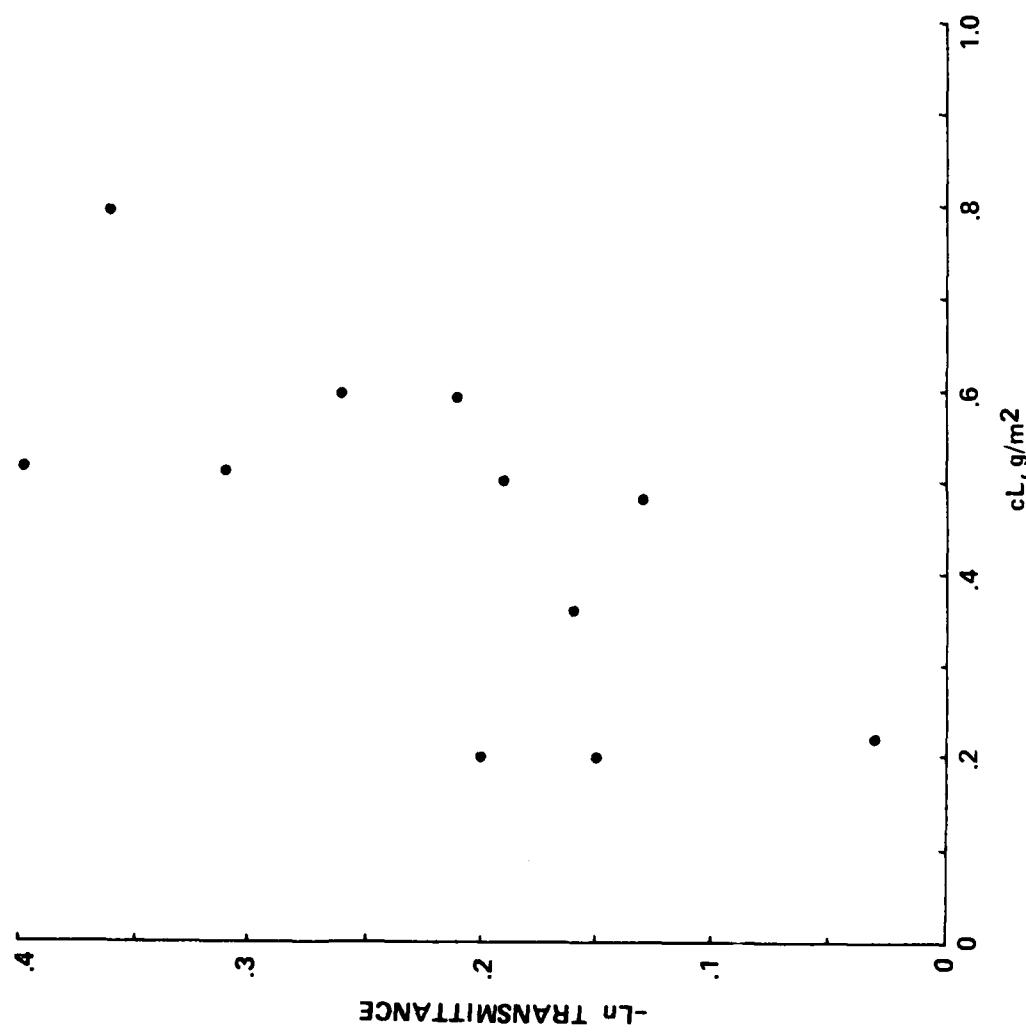
In the determination of the primary cause of this scatter in the data, the literature<sup>2</sup> gives a possible clue in the filter system flow rate. (The flow rate for each filter in the  $B_4C$  experiments was one liter/minute.) As described in Reference 2, there are two competing processes which determine the required flow rates in the filter collection system: the sedimentation due to gravity which removes particles from the air, and the inertia of the particles themselves which hinders them from following the curving flowstream to the collection filter. The flow rate must then be high enough to overcome the sedimentation effect but low enough for the particles to follow the flowstream.

To overcome the sedimentation due to gravity, the flow velocity through the collector must be much higher than the terminal velocity of a falling particle. On the other hand, flow rates and, consequently, flow velocities cannot be so high that particle inertia effects become serious. Solving the equations<sup>2</sup> (and assuming  $B_4C$  particles can be described as spheres) shows that the minimum flow rate must be greater than about one liter/minute for three micrometer diameter  $B_4C$  particles. The upper limit was calculated to be 500 liters/minute. Based on these considerations, equipment was modified to permit each of the two filter holders to have a 14 liter/minute flow rate.

---

2. Petracca, Carmine and Lindberg, James D., Installation and Operation of an Atmospheric Particulate Collector, Research and Development Technical Report ECOM-5575, Atmospheric Sciences Laboratory, U.S. Army Electronics Command, White Sands Missile Range, New Mexico 88002 (September 1975) AD #A016163. Available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, Virginia 22161.

FIGURE 13  
 $B_4C$  EXTINCTION  
AT 10.6 MICROMETERS



#### 4. SILICON CARBIDE (SiC) EXTINCTION COEFFICIENT AT 10.6 MICROMETERS

Silicon Carbide (Type 2 from Microabrasive Corporation, Westfield, Massachusetts) has the smallest commercially available particle size (2-3 micrometer diameter). Figure 14 shows a scanning electron micrograph of the particles. The bar at the bottom represents one micrometer.

Basically, the experimental arrangement in Figure 12 applies, except for the increased flow rate. The type of filter used was Millipore<sup>®</sup> Type PH, with .3 micrometer pore size. Good aerosol cloud homogeneity is indicated by the weights of SiC collected on the pairs of filters for each of the five runs (see Table 1). Figure 15 shows the plot of  $-\ln$  transmittance versus  $cL$ . The scatter in the data is much reduced compared to that in Figure 13, showing that, in fact, the flow rate was a primary cause of the problems. The extinction coefficient equals  $.37 \text{ m}^2/\text{g}$  at 10.6 micrometers. The B<sub>4</sub>C and SiC values offer no improvement over aerosols obtained from burning phosphorus, currently the best aerosol screening agent in use by the military.

Attention must be paid to the possibility of multiple scattering in aerosol optical measurements. If the optical thickness, which is the negative natural logarithm of the transmittance (the numerator in equation (1)), is greater than .1, then corrections should be made for multiple scattering.<sup>3</sup>

An examination of Figure 15 shows that the vertical scale, which is in fact the optical thickness, extends to .45. However, there is no discernible curvature of the data. Presumably, if significant multiple scattering existed above .1, such curvature would be present. Possibly, the rule of thumb given is meant to apply in those cases where the extinction is entirely due to scattering,<sup>3</sup> and none due to absorption.

---

3. Van de Hulst, H. C., Light Scattering by Small Particles, John Wiley & Sons, Inc., New York, New York (1957), p. 6.

FIGURE 14  
SCANNING ELECTRON MICROGRAPH OF SILICON CARBIDE PARTICLES

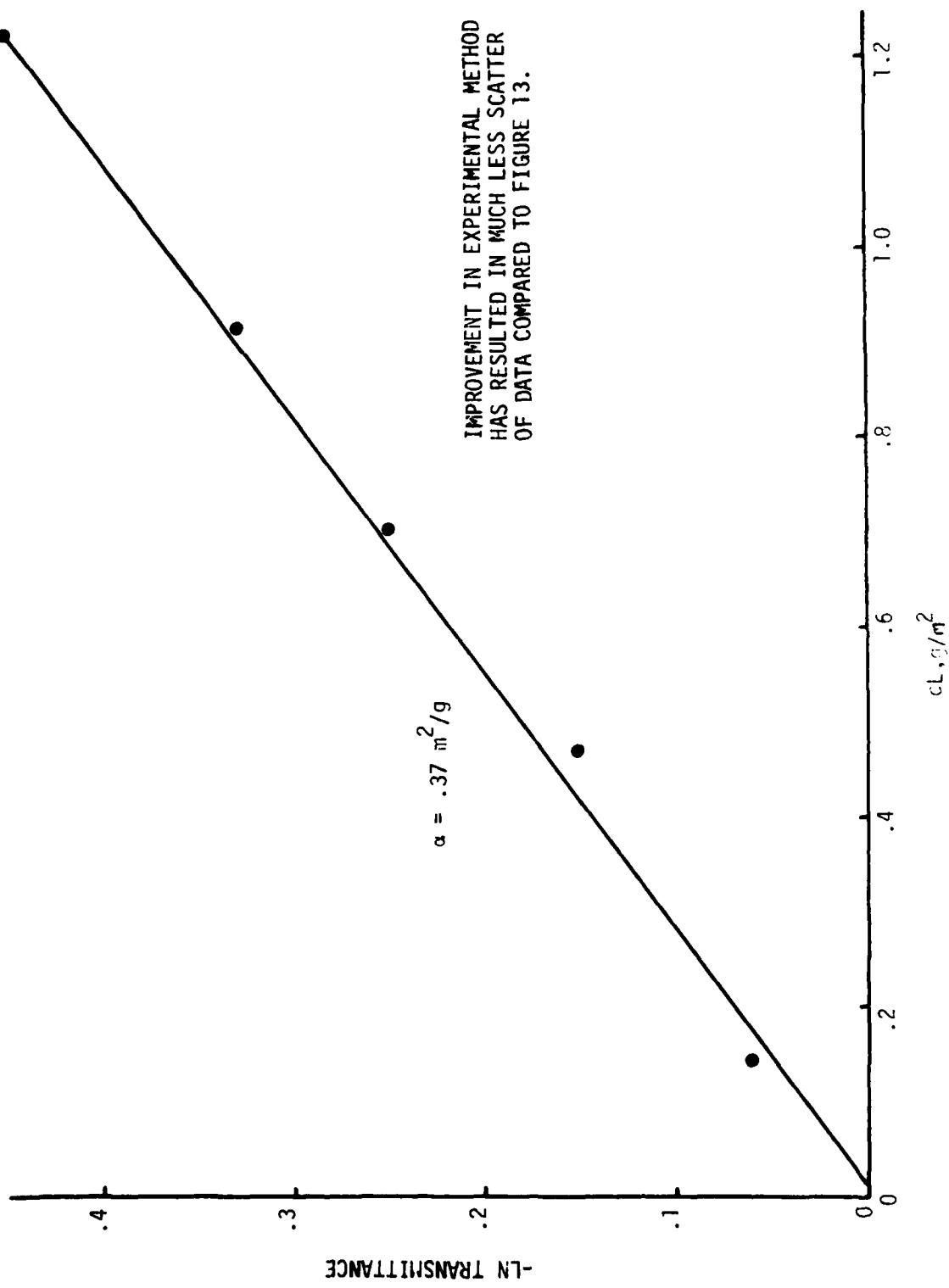


TABLE 1  
WEIGHTS OF SiC COLLECTED ON MILLIPORE<sup>®</sup> FILTER PAIRS,  
SHOWING AEROSOL HOMOGENEITY

<u>RUN</u>	<u>FILTER CLOSEST TO LASER<sup>†</sup></u>	<u>FILTER CLOSEST TO DETECTOR<sup>†</sup></u>
1	.0021 g	.0024 g
2	.0032	.0031
3	.0007	.0006
4	.0045	.0037
5	.0056	.0055

<sup>†</sup>See Figure 12

FIGURE 15  
SiC EXTINCTION AT  $10.6\mu\text{m}$



However, there is a non-negligible contribution by absorption to the extinction at 10.6 micrometers for  $B_4C$  and SiC, respectively.<sup>4,5</sup>

## 5. OLIVINE ( $Mg_2SiO_4$ )

In FY 78, a small sample of  $Mg_2SiO_4$  was obtained from Professor Don Huffman of the University of Arizona. Based on his measurements<sup>6</sup>, olivine has a strong extinction in the 10-micrometer region. In that work, olivine smoke was prepared by placing small amounts on a carbon electrode and striking an arc. The resulting smoke was collected on a glass slide and placed in an infrared spectrometer. Work at NAVWPNSUPPCEN Crane on olivine was directed toward making much larger quantities so that an aerosol can be formed in the chamber and laser extinction measurements made. A dual approach was pursued. One approach is to grind up olivine to create particles of 0.5 micrometer diameter<sup>7</sup>, which can then be dispersed using the dry powder disseminator. (The other approach, described later, is to pyrotechnically generate olivine.) The University of Arizona sample was ground, sieved, and ball milled to obtain a large fraction of particles less than one-micrometer diameter, as seen in Figure 16. The bar at the bottom is one micrometer. With this success, it was decided to grind and ball mill a few kilograms (kg) of olivine for use in the disseminator. Accordingly, about 3.5 kg of olivine (still in a basalt matrix) was received from Delmar Barker, a colleague of Don

---

4. Lauer, James L., and Adari, Hedy, "Optical Constants of Boron Carbide in the Infrared" in Basic Optical Properties of Materials, National Bureau of Standards Special Publication 574, Topical Conference on Basic Optical Properties of Materials, National Bureau of Standards, Gaithersburg, Maryland (May 5-7, 1980).

5. Huffman, Donald R., Determination of Optical Constants for Powdered Materials in the 10-Micron Infrared Region, Final Report, Department of Physics, University of Arizona, Tucson, Arizona 85721 (September 1979) Naval Air Systems Command Contract N00019-78-C-0479.

6. Huffman, D. R., Studies of Extinction by Small Particles in the 10-Micron Spectral Region, Final Report, Department of Physics, University of Arizona, Tucson, Arizona 85721 (July 1978) Naval Air Systems Command Contract N00019-77-C-0293.

7. Day, K. L., Steyer, T. R., and Huffman, D. R., A Quantitative Study of Silicate Extinction, Astrophys. J. 191, 415-418 (July 1974).

FIGURE 16

SCANNING ELECTRON MICROGRAPH OF OLIVINE PARTICLES GROUND, PASSED  
THROUGH A 300 MESH SIEVE AND BALL MILLED FOR 280 HOURS



Huffman. The rock was broken up, and as much basalt picked out by hand as possible. The olivine was ground and passed through a 300 mesh sieve. One hundred fifty milliliters (ml) of the material passing through the sieve (< 60 micrometer particle diameter) was placed in a new 15 cm ID by 15 cm high ceramic ball-milling crock with ten 1.9 cm diameter by 1.9 cm high ceramic cylinders. No fluids were added. After 64 hours of ball milling, 24 1.3 cm diameter steel balls were added. Ball milling was stopped after 159 hours. The olivine powder was of a loose fluffy consistency, and microscopic examination showed very few particles greater than two micrometers diameter.

Another 150 ml batch was ball milled, but this time, the olivine powder was found solidly caked along the side of the crock. Many further attempts were made to reproduce the first 150 ml batch, including carefully drying the -300 mesh olivine, the crock, the crock cover, and the cylinders and balls before milling. Sometimes a few percent fumed silica was added. Standard recommendations<sup>8</sup> were followed, all to no avail. In some instances, the particle size of the caked olivine seemed to be small enough, so attempts were made to salvage the olivine by scraping it out of the crock, breaking up lumps, and spreading the olivine out to a depth of two cm in a pan. The pan was placed in an oven at 100-120°C and the olivine dried for at least 15 hours. After 15 hours, there was no change. Either the temperature was not high enough to drive off any water present, or water was not responsible for the caking in the first place.

Because of the difficulties with ball milling olivine, attention was turned to attempts to pyrotechnically create olivine. The solid material magnesium silicide ( $Mg_2Si$ ) (Synthatron Corporation, Parsippany, New Jersey) was chosen<sup>9,10</sup> as the fuel, and common pyrotechnic materials such as alkali

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8. Jar, Ball and Pebble Milling, Theory and Practice, Bulletin P-291, Norton Company, Chemical Process Products Division, Akron, Ohio 44309.

9. Mellor, J. W., A Comprehensive Treatise on Inorganic and Theoretical Chemistry, Volume VI, John Wiley & Sons, Inc., New York, New York (1929).

10. Bailar, J. C., Jr.; Emeleus, H. J.; Nyholm, Sir Ronald; Trotman-Dickenson, A. F.; Comprehensive Inorganic Chemistry, Volume I, Pergamon Press, Oxford, UK (1973).

metal nitrates and perchlorates were selected as the oxidizers. The intent was to cause the reaction  $Mg_2Si + 2O_2$  (from oxidizer)  $\rightarrow Mg_2SiO_4$  to occur. A series of thermochemical calculations were run using the NASA Thermodynamics Program<sup>11</sup> to provide a starting point for composition formulation. Some results of these calculations are shown in Table 2. About 21%  $Mg_2SiO_4$  is the best that is predicted. This  $Mg_2SiO_4$  is predicted to be in liquid form. Amounts of solid  $Mg_2SiO_4$  were negligible. Very little gaseous species are present because the liquid boils at 4727°C, if indeed the gaseous species exists at all. It was not listed in the program output as a species being considered. If  $Mg_2SiO_4$  particles are formed smaller than about .5 micrometers, then Figure 17 shows that the possibility exists that these small particles may have a much higher extinction efficiency than particles produced by grinding and ball milling.

A series of hand-tamped 10-gram candles were constructed. (See Table 3.) Sodium nitrate was substituted for lithium nitrate, and potassium perchlorate was substituted for lithium perchlorate, to avoid hygroscopicity problems. Corrections were made in the formula percentages to allow for these changes.

The following are the results of burning the candles (based on 3-5 candles of each type). Optical measurements were not taken. The nomenclature is that followed in Table 3.

No. 1 - Somewhat difficult to ignite, 10 cm flame, medium vigor, little smoke, about 5 seconds burn time

No. 2 - Medium vigor, 15 cm flame, little smoke, 4-5 seconds burn time

No. 3 - Instant ignition, violent flame, about 1-3 seconds burn time, very little smoke

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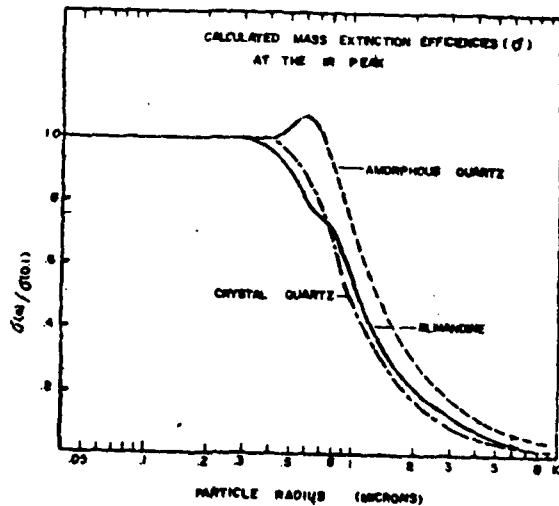
11. Gordon, S. and McBride, B. J., Computer Program for Calculation of Complex Chemical Equilibrium Compositions, Rocket Performance, Incident and Reflected Shocks, and Chapman-Jouguet Detonations, NASA SP-273, Lewis Research Center (1971). Available NTIS--N7137775.

TABLE 2  
RESULTS OF THERMODYNAMIC CALCULATIONS INVOLVING MAGNESIUM SILICIDE  
AND ALKALI METAL NITRATES AND PERCHLORATES

Fuel Percent	Equivalence Ratio	Wt. Percent of Mg <sub>2</sub> SiO <sub>4</sub> (liquid) <sup>t</sup>	Adiabatic Temperature, K
<u>Mg<sub>2</sub>Si/LiClO<sub>4</sub>:</u>			
40	.93	12	3234
50	1.34	12	3218
60	1.96	10	2807
<u>Mg<sub>2</sub>Si/LiNO<sub>3</sub>:</u>			
40	.97	21	3080
50	1.36	19	3094
55	1.63	17	2848

<sup>t</sup>No gas or solid phase Mg<sub>2</sub>SiO<sub>4</sub> is predicted by the NASA thermodynamics program.

FIGURE 17  
SILICATE EXTINCTION EFFICIENCIES  
(Taken from Reference 5)



The peak mass extinction efficiencies in the infrared for three silicates have been normalized to unity for spheres with radii equal to  $0.1 \mu$ . The efficiencies for particle sizes commonly produced by grinding (a few microns) are seen to be considerably below those of the smaller grains that have been proposed to occur in the inter- and circumstellar medium.

TABLE 3  
MAGNESIUM SILICIDE EXPERIMENTAL FORMULATIONS<sup>t</sup>, WEIGHT PERCENT

	<u>No. 1</u>	<u>No. 2</u>	<u>No. 3</u>	<u>No. 4</u>	<u>No. 5</u>
Mg <sub>2</sub> Si	25	35	45	37	45
NaNO <sub>3</sub>	65	55	45	38	-
Epoxy Binder <sup>tt</sup>	10	10	10	10	5
Olivine Powder	-	-	-	15	-
KC1O <sub>4</sub>	-	-	-	-	50

<sup>t</sup>Hand tamped into 1.2 cm inside diameter phenolic tubes of 1.8 cm length. All candles had about 10 g of composition.

<sup>tt</sup>Mixture of 70% DER 321 and 30% DEH 14 (Dow Chemical Corporation, Midland, Michigan).

No. 4 - Difficult to ignite, 10 cm flame, very little smoke

No. 5 - Medium vigor, 15 cm flame, little smoke, 4-5 seconds burn time

Since none of the compositions produced much smoke, further work on this was stopped.

## 6. RENOVATION/MODIFICATION OF CLOUD CHAMBER

Figure 18 shows a top view schematic of the cloud chamber facility being renovated and modified for infrared screening aerosol measurements. Extra support for this effort was provided by NAVAIR (Code 360D) under another task. One of the reasons that it is necessary to change to this location is that the experiments reported above in Sections 3 and 4 were done in the NAVWPNSUPPCEN Photometric Tunnel (Figure 12). This tunnel is normally used for testing flares on a regular basis, and the aerosol experiment arrangement is incompatible with this function. This means that after each aerosol experiment was done, all equipment had to be completely removed, which is very time consuming. The new facility, totally dedicated to aerosol experiments, will solve this problem.

The original structure is the cloud chamber itself at A. The dimensions are 2.5 m high by 3 m long by 3 m wide. An instrument room at B is also part of the original structure. A trailer has been added at C, and a new room has been added at D. These new additions will allow access to two more walls of the chamber, enabling more aerosol instrumentation to be used in experiments.

The CO<sub>2</sub> laser, detector, and filters that had been indicated in Figure 12 have been moved from that location to the present one. These instruments are now located at B1, A1, and A2 in Figure 18. Also indicated in Figure 18 (but not in Figure 12) at A3 is the pump for the filter system. Figure 19 shows an outside view. The letters A, B, and C have the same meaning as in Figure 18. When this picture was taken, room D had not yet been built.

FIGURE 18  
NEW AEROSOL MEASUREMENT FACILITY

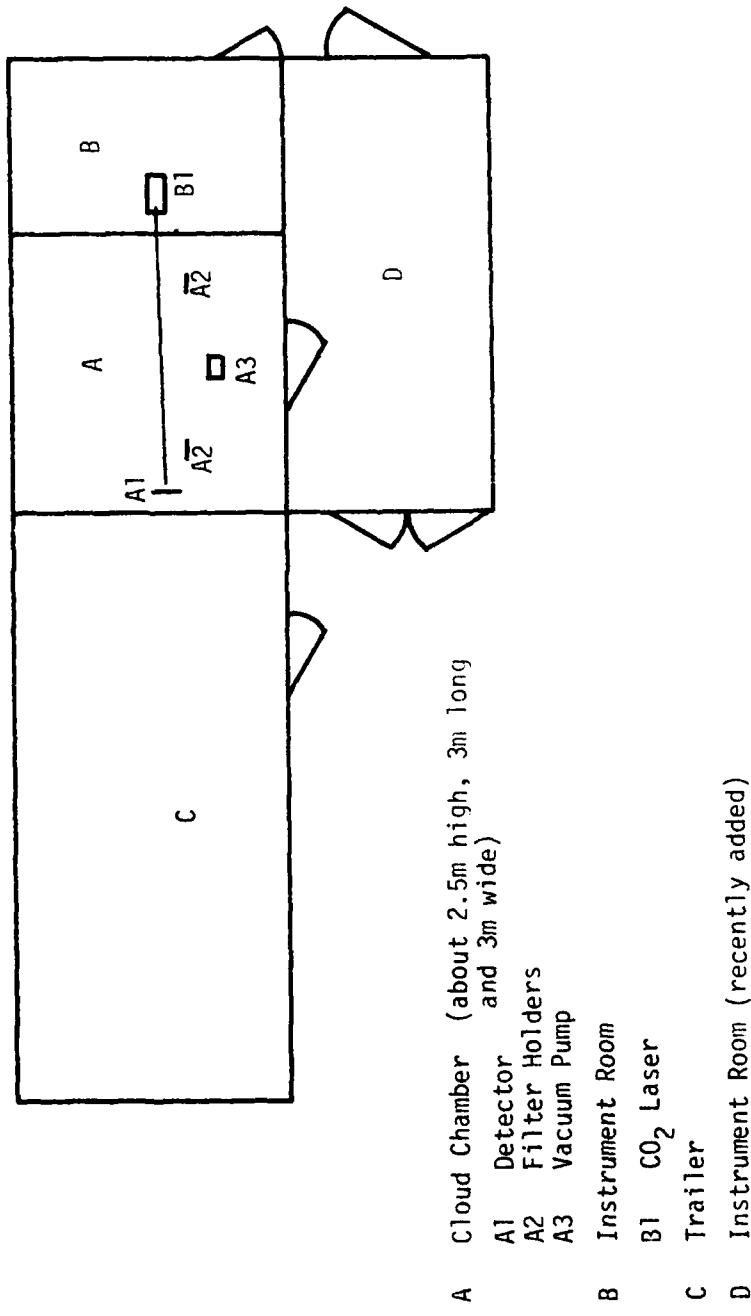


FIGURE 19. OUTSIDE VIEW OF NEW AEROSOL MEASUREMENT FACILITY

- A is Cloud Chamber
- B is Instrument Room
- C is Trailer (new instrument room not built at time this photo was taken)

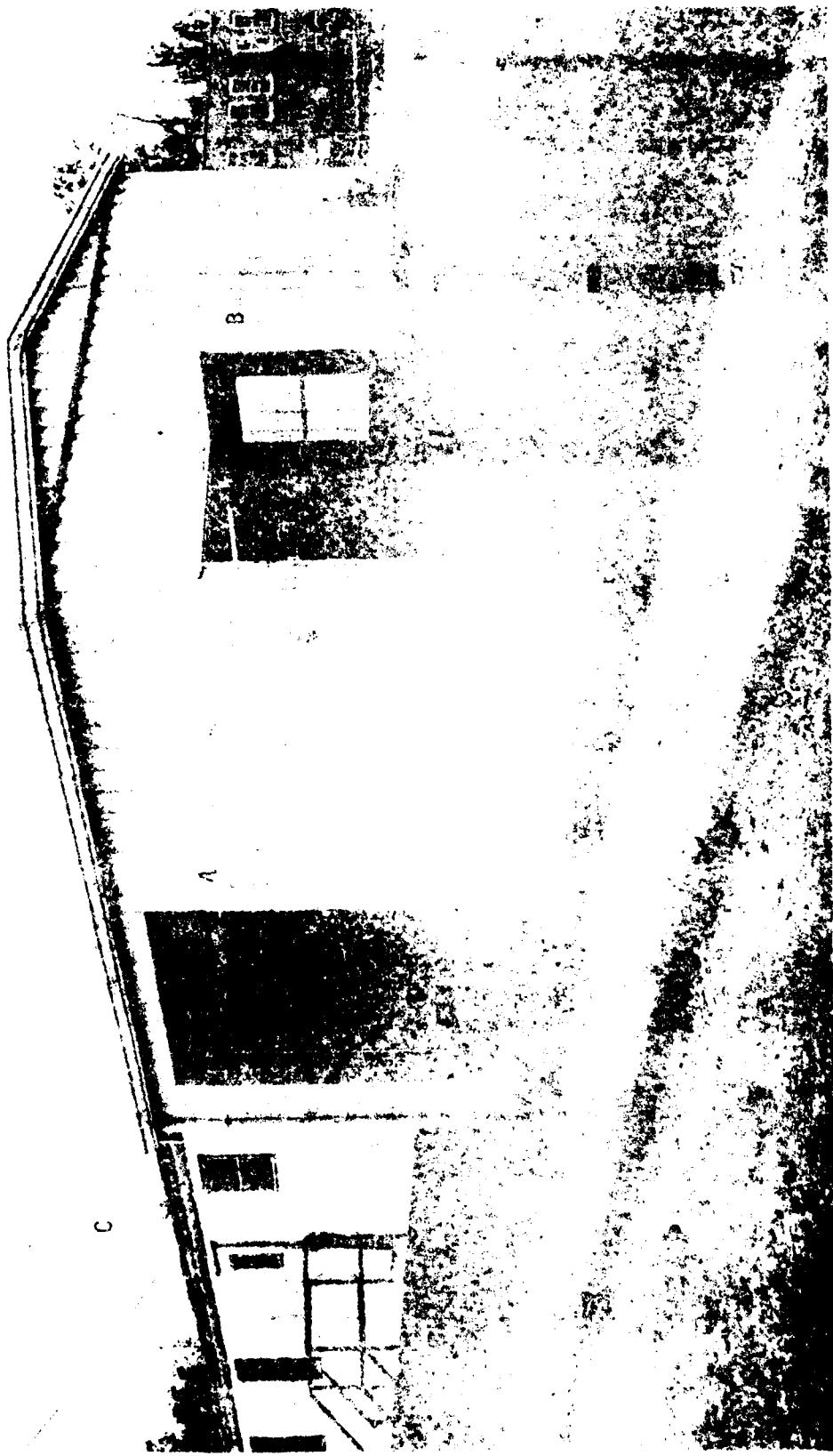
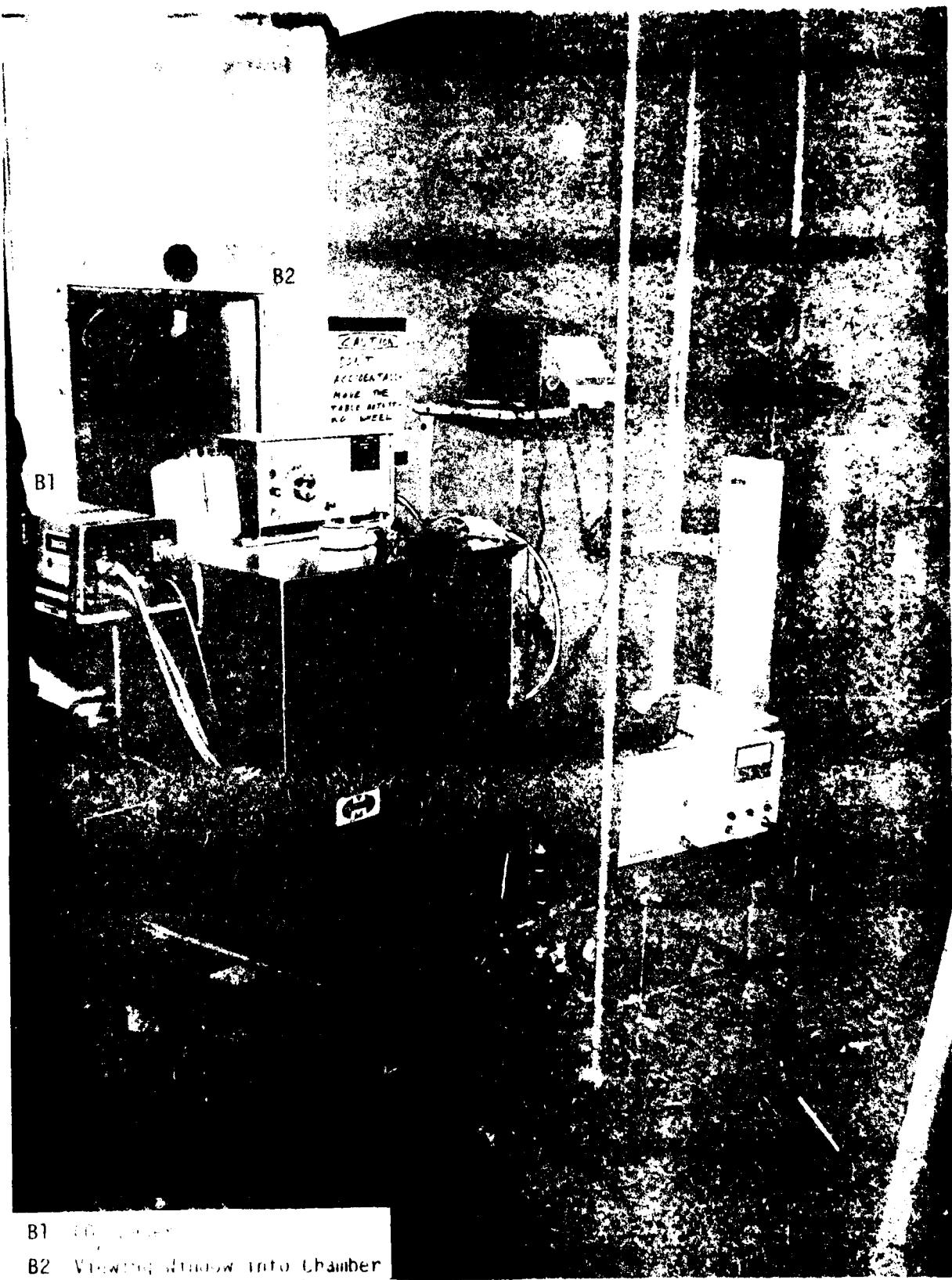


Figure 20 shows the instrument room "B." The CO<sub>2</sub> laser is shown at B1. A viewing window into the cloud chamber can be seen at B2. Because of existing nonremovable equipment associated with the original function as a cloud chamber, this room is extremely crowded. Figure 21 shows part of the interior of the chamber. The CO<sub>2</sub> laser optical path is indicated by a dashed line which passes about 15 cm in front of one of the filter holders at A2 (see Figure 18 for the optical layout). The detector is off to the left, out of the photo, while the other filter holder is off to the right, also out of the photo. The (plastic-covered) filter pump is shown on the floor at A3. The vertical rod in front of the pump is used to support the aerosol disseminator (Figure 9). Figure 22 is another interior photo, taken to the left of Figure 21. Now the detector can be seen at A1 and the same filter as in Figure 21 is barely seen at A2. Again, the optical axis is indicated by a dashed line.

When the renovation of the facility is complete, there will be enough room to include other in-house instruments, such as the Whitby Aerosol Analyzer (TSI, Inc., St. Paul, Minnesota), Knollenberg Particle Size Analyzer (PMS, Inc., Boulder, Colorado), Andersen Impactor (Andersen 2000, Inc., Atlanta, Georgia), a Nd:YAG 1.06 micrometer laser (General Photonics, Inc., Mountain View, California), and either an infrared spectrometer or a circular variable filter system.

FIGURE 20. INSTRUMENTATION.



B1 Control Panel

B2 Viewing Window into Chamber

FIGURE 10 - PLATE INTERIOR - 100% FILTER



A3 - water pump for filters



A1 is

A2 is

Dashed

dx15

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